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Communication

NONCONVENTIONAL FRIEDEL-CRAFTS CHEMISTRY III. ON THE REGIOSPECIFIC SYNTHESIS OF SPIRO[CYCLOALKANE-1,1'-ISOTHIOCHROMAN]-4'-ONE DERIVATIVES

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The reaction of 1-oxa-4-thiaspiro[4,4]nonan-2-one (**4**) and/or 1-oxa-4-thiaspiro[4,5]decan-2-one (**5**) with arenes (**6**) under the catalytic action of aluminum chloride afforded in all cases spiro[cycloalkane-1,1'-isothiochroman]-4'-ones (**7a-g** and **8a-g**), [(arylcycloalkyl)thio]-acetic acid, (**9a-g** and **10a-g**), cycloalkylthioacetic acids, (**11** and **20**) aryl cycloalkyl sulfides, (**14** and **23**) diaryl sulfides (**15**), diaryl disulfides and dicycloalkyl disulfides (**13** and **22**). The mechanisms of these reactions are discussed.

Key words: Spiroisothiochroman-4-ones; Friedel-Crafts reactions.

INTRODUCTION

In continuation to our interest in sulfur and Friedel-Crafts chemistry,¹⁻¹⁰ the use of Friedel-Crafts reactions in the synthesis of sulfur compounds,¹¹⁻¹⁹ together with the importance of spirocycles,²⁰⁻⁴⁰ this work presents a facile route for the synthesis of some spiro[cycloalkane-1,1'-isothiochroman]-4'-ones. These syntheses were based on the structural similarities between the lactones and 1-oxa-4-thiaspiro[4,4]nonan-2-one and 1-oxa-4-thiaspiro[4,5]decan-2-one.

RESULTS AND DISCUSSIONS

Our precursors, 1-oxa-4-thiaspiro[4,4]nonan-2-one (**4**) and 1-oxa-4-thiaspiro[4,5]decan-2-one (**5**) were prepared according to literature procedure,⁴¹ starting with the commercially available cyclopentanone (**1**), and/or cyclohexanone (**2**) which were reacted with mercaptoacetic acid (**3**) in the presence of *p*-toluene sulfonic acid catalyst (Equation 1).

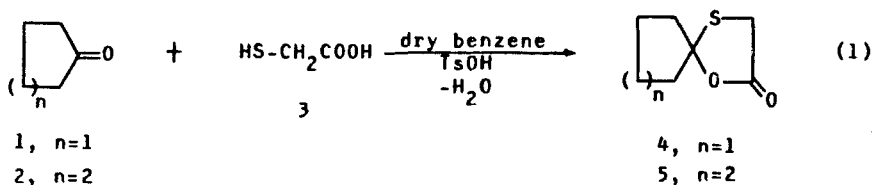


TABLE I

Reactions of 1-oxa-4-thiaspiro[4,4]nonan-2-one and/or 1-oxa-4-thiaspiro[4,5]decan-2-one with various arenes in the presence of AlCl_3 catalyst^a

Entry	Arene	1-O/Arene/Catalyst ^b	Observed products ^c (%)
1	benzene	0.01/25/0.044	spiro[cyclopentane-1,1'-isothiochroman]-4'-one ^d (60), [[1-phenyl-cyclopentyl]thio]acetic acid (15), cyclopentylthioacetic acid (7), cyclopentylmercaptane (3), dicyclopentyl disulfide (3), cyclopentyl phenyl sulfide (5), diphenyl sulfide (2).
2	toluene	0.01/25/0.044	6'-methylspiro[cyclopentane-1,1'-isothiochroman]-4'-one (78), [[1-(4-tolyl cyclopentyl)]thio]acetic acid (8), cyclopentylthioacetic acid (6), cyclopentyl mercaptane (3), dicyclopentyl disulfide (2), ditolyl sulfide (3).
3	<i>p</i> -xylene	0.01/25/0.044	5',8'-dimethylspiro[cyclopentane-1,1'-isothiochroman]-4'-one (80), [[1-(2',5'-dimethylphenyl)cyclopentyl]thio]acetic acid (5), cyclopentyl mercaptane (2), dicyclopentyl disulfide (2), dixylyl sulfide (4), cyclopentylthioacetic acid (3).
4	bromobenzene	0.01/25/0.044	6'-bromospiro[cyclopentane-1,1'-isothiochroman]-4'-one (50), [[1-(4-bromophenyl)cyclopentyl]thio]acetic acid (15), cyclopentylthioacetic acid (10), cyclopentyl mercaptane (5), dicyclopentyl disulfide (3), 4-bromophenyl cyclopentyl sulfide (2).
5	chlorobenzene	0.01/25/0.044	6'-chlorospiro[cyclopentane-1,1'-isothiochroman]-4'-one (50), [[1-(4-chlorophenyl)cyclopentyl]thio]acetic acid (15), cyclopentylthioacetic acid (7), cyclopentyl mercaptane (3), 4-chlorophenyl cyclopentyl sulfide (3), dicyclopentyl disulfide (4).
6	anisole	0.01/25/0.044	6'-methoxyspiro[cyclopentane-1,1'-isothiochroman]-4'-one (55), 6'-hydroxyspiro[cyclopentane-1,1'-isothiochroman]-4'-one (15), [[1-(4-methoxyphenyl)cyclopentyl]thio]acetic acid (5), [[1-(4-hydroxyphenyl)cyclopentyl]thio]acetic acid (5), cyclopentylthioacetic acid (3), cyclopentyl mercaptane (2), dicyclopentyl disulfide (3), dianisyl sulfide (4).
7	biphenyl ^e	0.01/0.02/0.044	6'-phenylspiro[cyclopentane-1,1'-isothiochroman]-4'-one (76), [[1-(biphenyl)cyclopentyl]thio]acetic acid (5), cyclopentylthioacetic acid (4), cyclopentylmercaptane (3), dicyclopentyl disulfide (2), biphenyl cyclopentyl sulfide (2).
8	benzene	0.01/25/0.044	spiro[cyclohexane-1,1'-isothiochroman]-4'-one (70), [[1-(phenyl)-cyclohexyl]thio]acetic acid (6), cyclohexylthioacetic acid (5), cyclohexyl mercaptane (2), dicyclohexyl disulfide (3), cyclohexyl phenyl sulfide (2).
9	toluene	0.01/25/0.044	6'-methylspiro[cyclohexane-1,1'-isothiochroman]-4'-one (75), [[1-(4-tolyl)cyclohexyl]thio]acetic acid (7), cyclohexylthioacetic acid (3), cyclohexyl mercaptane (2), dicyclohexyl disulfide (2), cyclohexyl 4-tolyl sulfide (2).
10	<i>p</i> -xylene	0.01/25/0.044	5',8'-dimethylspiro[cyclohexane-1,1'-isothiochroman]-4'-one (80), [[1-(2',5'-dimethylphenyl)cyclohexyl]thio]acetic acid (5), cyclohexylthioacetic acid (4), cyclohexyl mercaptane (2), dicyclohexyl disulfide (3), dixylyl sulfide (2).
11	bromobenzene	0.01/25/0.044	6'-bromospiro[cyclohexane-1,1'-isothiochroman]-4'-one (55), [[1-(4-bromophenyl)cyclohexyl]thio]acetic acid (10), cyclohexylthioacetic acid (5), cyclohexyl mercaptane (3), dicyclohexyl disulfide (3), 4-bromophenyl cyclohexyl sulfide (2).

TABLE I (Continued)

Entry	Arene	1-O/Arene/Catalyst ^b	Observed products ^c (%)
12	bromobenzene	0.01/25/0.044	6'-chlorospiro[cyclohexane-1,1'-isothiochroman]-4'-one (50), [[1-(4-chlorophenyl)cyclohexyl]thio]acetic acid (15), cyclohexylthioacetic acid (7), cyclohexyl mercaptane (4), 4-chlorophenyl cyclohexyl sulfide (2), dicyclohexyl disulfide (2).
13	anisole	0.01/25/0.044	6'-methoxyspiro[cyclohexane-1,1'-isothiochroman]-4'-one (70), 6'-hydroxyspiro[cyclohexane-1,1'-isothiochroman]-4'-one (10), [[1-(4-methoxyphenyl)cyclohexyl]thio]acetic acid (5), [[1-(4-hydroxyphenyl)cyclohexyl]thio]acetic acid (2), cyclohexylthioacetic acid (2), cyclohexyl mercaptane (2), dianisyl sulfide (2).
14	biphenyl ^d	0.01/0.02/0.044	6'-phenylspiro[cyclohexane-1,1'-isothiochroman]-4'-one (70), [[1-(biphenyl)cyclohexyl]thio]acetic acid (5), cyclohexylthioacetic acid (4), cyclohexyl mercaptane (3), dicyclohexyl disulfide (2), biphenyl cyclohexyl sulfide (2).

^a All the experiments have been carried out at room temperature and magnetically stirred for 24 hrs.

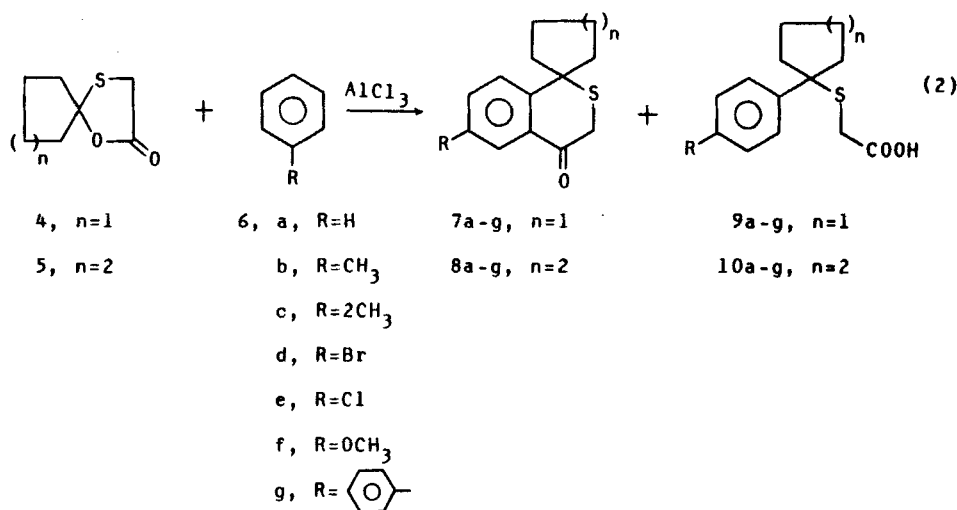
^b 1-O = 1-Oxa-4-thiaspiro[4,4]nonan-2-one and/or 1-Oxa-4-thiaspiro[4,5]decan-2-one. Units: 1-O, moles; arene, milliliters; catalyst, moles.

^c The percentages of these products were calculated by HPLC; the separations of these products were performed by preparative thin layer chromatography.

^d The nomenclature of these products was directed by Prof. Dr. Kurt L. Leoning, Managing Director of Topterm, North American Division, P. O. Box 21213, Columbus, Ohio 43221, U.S.A.

^e This experiment was carried out in CS₂ as a solvent; the excess biphenyl was removed by fractional crystallization.

Surveying the results of Table I showed that, in the presence of AlCl₃ catalyst, compound 4 and/or 5 reacted with arenes to yield spiro[cycloalkane-1,1'-isothiochroman]-4'-ones in good yields (50–80%), together with [(1-(aryl)cycloalkyl)thio]acetic acids (5–15%), in addition to other Friedel-Crafts products (Equation 2).

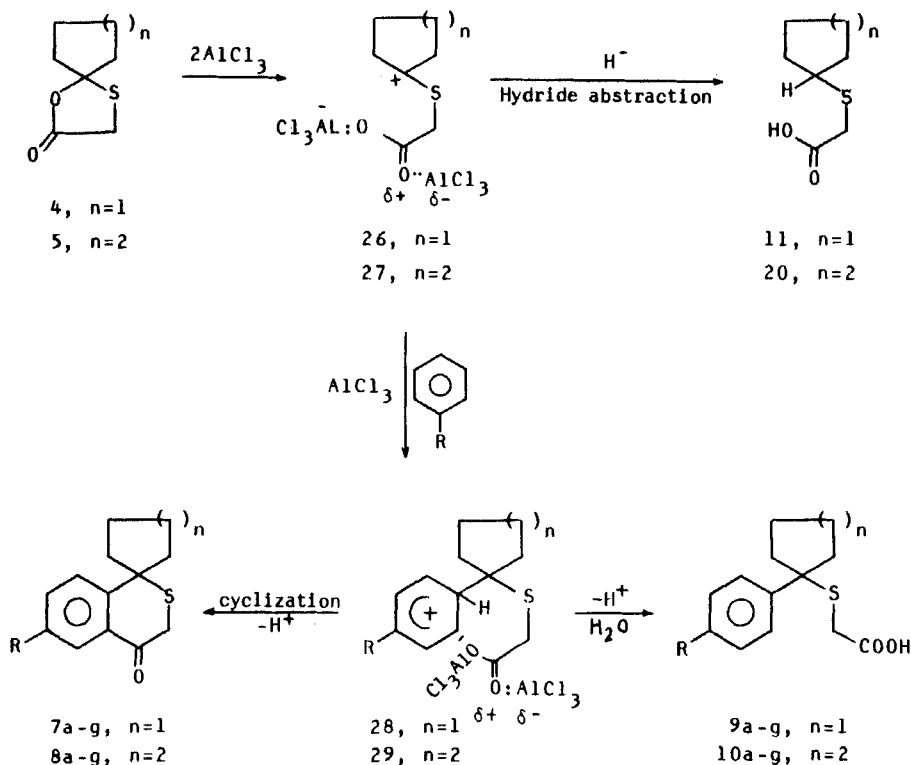


The formation of compounds **7a–g**, **8a–g**, **9a–g**, and **10a–g** could be explained⁴² as shown in Scheme I.

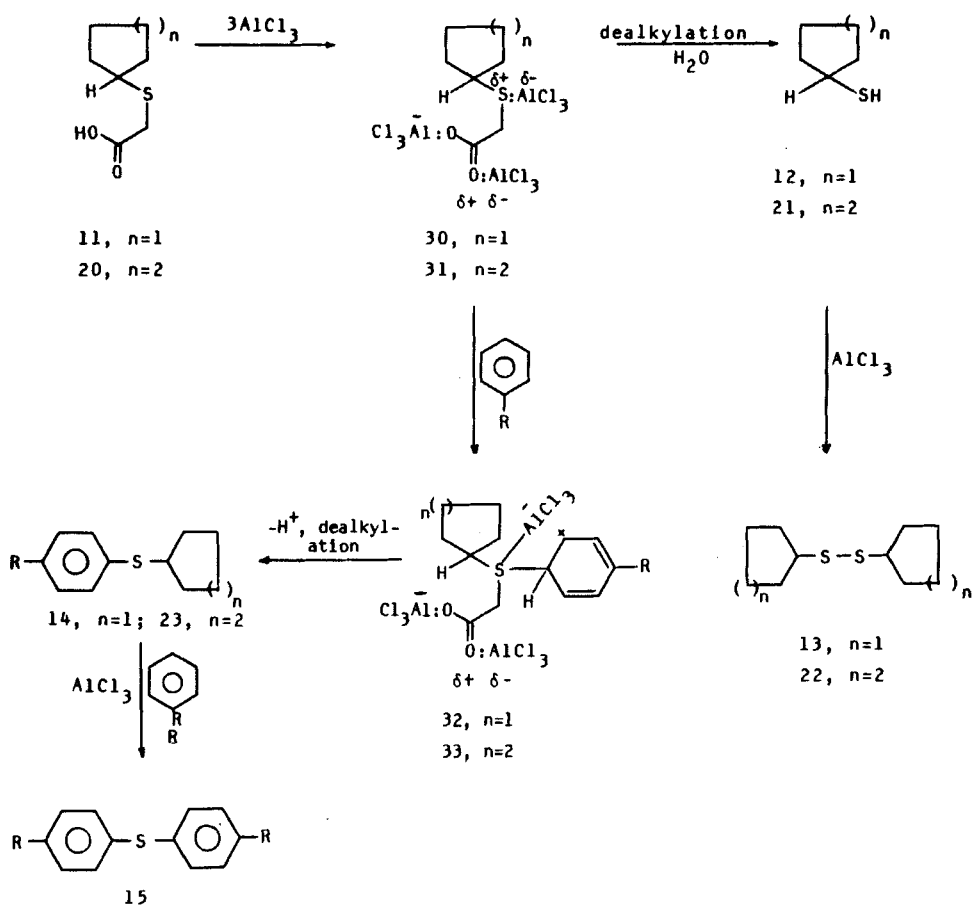
It is clear from Scheme I, that 1-oxa-4-thiaspiro[4,4]nonan-2-one (**4**) and/or 1-oxa-4-thiaspiro[4,5]decan-2-one (**5**) can be ionized by the Lewis acid catalyst to form the ion pairs **26** and **27**. The intermediates **26** and/or **27** can be arylated with arenes in the presence of AlCl_3 to yield **28** and/or **29** which are cyclized to **7a–g** and/or **8a–g**. Hydrolysis of **28** and/or **29** yielded the corresponding arylcycloalkylthioacetic acids **9a–g** and **10a–g**. Also **26** and/or **27** undergo hydride abstraction reactions⁴³ to afford the corresponding cycloalkylthioacetic acid **11** and **20** respectively.

The formation of cycloalkyl mercaptanes **12** and **21**, dicycloalkyl disulfides **13** and **22**, aryl cycloalkyl sulfides **14**, **23** and the diaryl sulfides **15** could be discussed^{6,8,44} as depicted in Scheme II.

Scheme II describes the complexation of **11** and/or **20** with AlCl_3 through the sulfur atom to give **30** and/or **31** which upon dealkylation afforded cyclopentyl mercaptane (**12**) and/or cyclohexyl mercaptane (**21**) respectively. Compounds **12** and **21** were oxidized to the corresponding disulfides **13** and **22** under the effect of AlCl_3 catalyst. Furthermore, the coordinated complexes **30** and **31** can be attacked by arenes to give tetravalent sulfur intermediates,⁴⁴ **32** and **33** which dealkylated under the mentioned reaction conditions to yield compounds **14** and **23**.



Scheme I



Scheme II

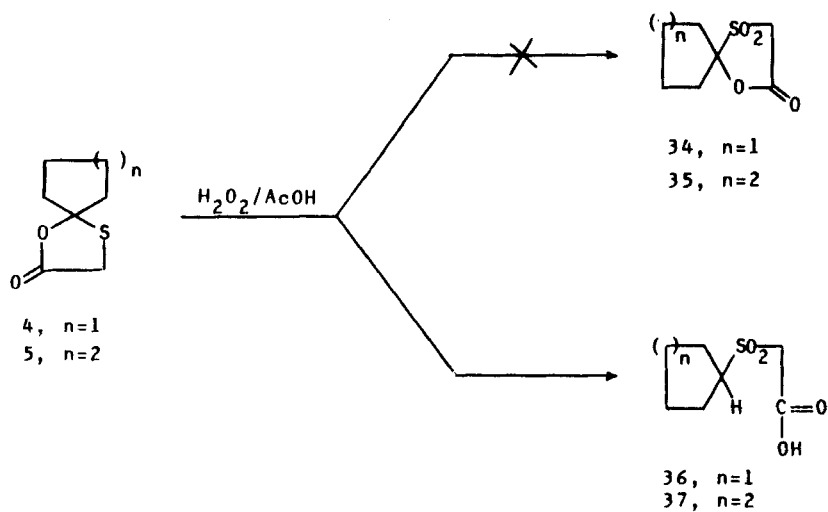
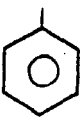
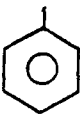


TABLE II
Physical Data of Spiro[cycloalkane-1,1'-isothiochroman]-4'-ones **7a-g** and **8a-g**.

Compd. No.	R	Yield ^a (%)	Molecular formula ^b (solvent of crystallization)	m.p. °C	IR (KBr), cm ⁻¹	¹ H NMR (solvent), δ (TMS) ppm
7a	H	60	C ₁₃ H ₁₄ OS (ethanol)	49-50	700 (C-S), 1700 (C=O), 2820 (CH aliph), 3050 (CH arom).	(CDCl ₃) 1.3-1.7 (4H, m), 1.8-2.3 (4H, m), 3.4 (2H, s), 7-7.6 (4H, m).
7b	CH ₃	78	C ₁₄ H ₁₆ OS (ethanol)	80-83	700 (C-S), 1700 (C=O), 2790-3010 (CH aliph), 3080 (CH arom).	(CDCl ₃) 1.3-1.6 (4H, m), 1.7-2.0 (4H, m), 2.3(3H, s), 3.45 (2H, s), 7-7.3 (2H, d), 7.5 (1H, s).
7c	2CH ₃	80	C ₁₅ H ₁₈ OS (ethanol)	90-93	700 (C-S), 1710 (C=O), 2860 (CH aliph), 3060 (CH arom).	(Acetone D ₆) 1.7-1.9 (4H, m), 2-2.1 (4H, m), 2.3 (6H, s), 3.27 (2H, s), 6.9-7.1 (2H, d).
7d	Br	50	C ₁₃ H ₁₃ OSBr (methylene chloride/ petroleum ether)	187-190	700 (C-S), 1712 (C=O), 2795 (CH aliph), 3060 (CH arom).	(CDCl ₃) 1.3-1.41 (4H, m), 1.66-2.1 (4H, m), 3.4 (2H, s), 7.2 (2H, d), 7.42 (1H, s).
7e	Cl	50	C ₁₃ H ₁₃ OSCl (ethanol/water)	68-70	900 (C-Cl), 700 (C-S), 1710 (C=O), 2850 (CH aliph), 3080 (CH arom).	(CDCl ₃) 1.4-1.55 (4H, m), 1.9-2.2 (4H, m), 3.4 (2H, s), 7-7.2 (2H, d), 7.4 (1H, s).
7f	OCH ₃	55	C ₁₄ H ₁₆ O ₂ S (ethanol)	70-73	700 (C-S), 1715 (C=O), 2860 (CH aliph), 3080 (CH arom).	(Acetone D ₆) 1.2-1.5 (4H, m), 1.59-1.70 (4H, m), 3.4 (2H, s), 3.6 (3H, s), 6.6-6.8 (2H, d), 6.9 (1H, s).
7g		76	C ₁₉ H ₁₈ OS (methanol)	87-90	710 (C-S), 1700 (C=O), 2890 (CH aliph), 3030-3080 (CH arom).	(Benzene D ₆) 1.3-1.5 (4H, m), 1.7-2.1 (4H, m), 3.42 (2H, s), 7.3-7.6 (8H, complex).
8a	H	70	C ₁₄ H ₁₆ OS (ethanol)	58-60	700 (C-S), 1710 (C=O), 2860 (CH aliph), 3040 (CH arom).	(Benzene D ₆) 1.3-1.4 (6H, complex), 1.7-2 (4H, complex), 3.3 (2H, s), 7.1-7.4 (5H, complex).
8b	CH ₃	75	C ₁₅ H ₁₈ OS (ethanol/water)	115-117	700 C-S), 1710 (C=O), 2870 (CH aliph), 3050 (CH arom).	(CDCl ₃) 1.3-1.4 (6H, complex), 1.7-2 (4H, complex), 2.23 (3H, s), 3.4 (2H, s), 6.7-6.9 (2H, d), 7.1 (1H, s).
8c	2CH ₃	80	C ₁₆ H ₂₀ OS (ethanol)	250-252	700 (C-S), 1715 (C=O), 2870 (CH aliph), 3060 (CH arom).	(Acetone D ₆) 1.23-1.4 (6H, complex), 1.67-1.9 (4H, complex), 2.24 (6H, s), 3.1 (2H, s), 6.8 (2H, d).
8d	Br	55	C ₁₄ H ₁₅ OSBr (ethanol)	150-152	700 (C-S), 1710 (C=O), 2860 (CH aliph), 3080 (CH arom).	(CDCl ₃) 1.27-1.55 (6H, complex), 1.7-1.9 (4H, complex), 3.4 (2H, s), 6.7-6.9 (2H, d), 7.1 (1H, s).

8e	Cl	50	$C_{14}H_{15}OSCl$ (methanol)	108–110	700 (C–S), 1710 (C=O), 2870 (CH aliph), 3060 (CH arom).	(CDCl ₃) 1.29–1.55 (6H, complex), 1.71– 1.9 (4H, complex), 3.23 (2H, s), 6.76–6.9 (2H, d), 7.12 (1H, s).
8f	OCH ₃	70	$C_{15}H_{10}O_2S$ (methanol)	160–162	700 (C–S), 1710 (C=O), 2860 (CH aliph), 3080 (CH, arom).	(Acetone D ₆) 1.25–1.55 (6H, complex), 1.65–1.8 (4H, complex), 3.5 (3H, s), 3.8 (2H, s), 6.67–6.9 (2H, d), 7.1 (1H, s).
8g		70	$C_{20}H_{20}OS$ (ethanol/water)	125–127	710 (C–S), 1715 (C=O), 2865 (CH aliph), 3070 (CH arom).	(CF ₃ COOH) 1.3–1.5 (6H, complex, 1.71–1.9 (4H, complex), 3.4 (2H, s), 7.3–7.6 (8H, complex).

^a The percentages of yields were calculated by HPLC.

^b Compounds **7a–g** and **8a–g** gave satisfactory elemental analysis.

The formation of compounds **15a–g** results from the subsequent alkylation of **14** and **23** with the solvent in the presence of AlCl_3 .⁴⁴

The conversion of **4** and **5** to the ion pairs **26** and **27** under the effect of acidic catalyst was elaborated through our trails for the oxidation of **4** and **5** using hydrogen peroxide in glacial acetic acid, instead of the expected cyclic spiro-sulfones **34** and **35**, the corresponding cycloalkylsulfonylacetic acids **36** and **37** were formed (Equation 3). All the formed spiro[cycloalkane-1,1'-isothiochroman]-4'-ones **7a–g** and **8a–g** and arylcycloalkylthioacetic acid **9a–g** and **10a–g** were identified by conventional methods (IR, ^1H -NMR and elemental analyses) (Table II).

Other products, cycloalkylthioacetic acid, cycloalkyl mercaptanes, dicycloalkyl disulfides, aryl cycloalkyl sulfides and diaryl sulfides were identified with HPLC technique using authentic samples.

Summing up, this paper can offer facile routes for the synthesis of regiospecific spiran derivatives of isothiochroman-4-ones.

EXPERIMENTAL

Melting points are uncorrected. Nuclear magnetic spectra were measured on EM-360 90 MHz spectrophotometer. Infrared spectra were recorded on a Pye-Unicam SP 200-G spectrophotometer. Isolation of products was achieved on a 20×15 glass plate covered with thin silica gel film. UV absorbances were measured on a Perkin-Elmer 552 spectrophotometer. Elemental analyses were recorded on a Perkin-Elmer 240 C microanalyser.

High-pressure liquid chromatography (HPLC) analyses were performed on a Hitachi apparatus (Japan) supplied with a variable wavelength monitor (190–600 nm), with a sulfonated silica gel type column, Nucleosil 55A, and methanol/water mixtures as a solvent.

Reaction of 1-oxa-4-thiaspiro[4,4]nonan-2-one (4) and/or 1-oxa-4-thiaspiro[4,5]decan-2-one (5) with arenes in the presence of AlCl_3 catalyst.

General Procedure. A sample of 0.044 mol of AlCl_3 was added to a solution of 0.01 mol of **4** or **5** in 25 ml of the arene in a two-necked flask equipped with a reflux condenser capped with calcium chloride tube, a magnetic stirrer, and a dropping funnel. The reaction mixture was stirred for 24 h at room temperature, decomposed with 10% HCl solution, and extracted with chloroform and methylene chloride; the combined extracts were washed with water, 10% sodium carbonate solution, and again water and dried over magnesium sulfate. The alkaline wash was acidified, then extracted with methylene chloride. The solvents and the unreacted arene were removed by distillation under reduced pressure on a rotary evaporator, and the residue was subjected to HPLC analyses and to separation and purification by preparative thin layer chromatography. Products were identified as described under each individual run. Results are found in Table I and the physical data of products **7a–g** and **8a–g** is depicted in Table II.

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